

INCIDENCE, CONCENTRATION AND EFFECTIVENESS OF MALATHION RESIDUES IN WHEAT AND MAIZE (CORN) EXPORTED FROM THE UNITED STATES

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Abstract—The incidence, concentration, and effectiveness of malathion treatments were measured in wheat and maize samples obtained from 79 port terminals in the United States. The presence of biologically active residue levels of malathion was inferred by bioassay assessment in 28% of 2058 wheat samples and in 8.4% of 2383 maize samples examined during the 2-year period, January 1977–December 1978. Malathion occurred most often in wheat from port terminals in the Pacific and Gulf regions where treatments were frequently part of the grain sale agreement and where grain was often treated immediately prior to sampling. At terminals where the grain was not treated just prior to sampling, the incidence of malathion on grain arriving at the ports was only 11.6% for wheat and 7.7% for maize.

Average malathion residues were 2.50 ppm in 210 samples of wheat and 1.03 ppm in 139 samples of maize and ranged from 2.71 ppm in Gulf wheat to 0.66 ppm in Pacific maize. Residues on wheat treated just prior to sampling, show inconsistent and generally inadequate malathion deposits resulting from port terminal treatments. No consistent relationship was evident between grain handling capacities and malathion residues.

INTRODUCTION

THE ORGANOPHOSPHORUS compound, malathion, is the only readily available chemical grain protectant presently approved in the United States for direct application to grain (STOREY *et al.*, 1979). It has been recommended for application to stored grain for nearly 20 years. Resistance in some stored grain insects to malathion has been reported in many areas of the world by DYTE (1974) and further documented among strains of *Tribolium castaneum* (Herbst) in U.S. peanuts (ZETTLER, 1974), rice (ZETTLER, 1975) and farm storages and flour mills in North Carolina (BANSODE and CAMPBELL, 1979). Resistance to malathion was also reported in several strains of *Cadra cautella* (Walker) and *Plodia interpunctella* (Hubner) collected from peanut- and grain-storage facilities (ZETTLER *et al.*, 1973). Irrespective of such data, relatively little information is available to indicate the extent of malathion use in the U.S. grain marketing system or its overall effectiveness.

Studies presented here were developed to show a national overview of the incidence, concentration and effectiveness of malathion on wheat and maize (corn) passing through export terminals in the U.S. This study was conducted in conjunction with a national survey to determine the incidence of insect infestation in wheat and maize exported from the United States by STOREY *et al.* (1982).

MATERIALS AND METHODS

Grain samples

Grain samples for malathion and insect analyses were obtained from randomly selected sublots of wheat and maize (corn) loaded on ships at 79 port elevators in the United States and certain Canadian ports in the St. Lawrence Seaway during the 2-year

period from January 1977 to December 1978. All samples were obtained by diverter-type mechanical grain samplers. These devices are installed at the end of a conveyer belt or within a spout and draw their sample for official grade analysis by periodically moving a pelican-like device through the entire grain stream. The collected sample is reduced in size by passing the grain through a divider or secondary sampler and then delivered to a collection box or sample bucket. About 1 kg of this sample was sent to the U.S. Grain Marketing Research Laboratory for analysis. When received, each sample was catalogued by elevator location, sample number, commodity, and the dates samples were received. A total of 2058 samples of wheat and 2383 samples of maize were examined.

During the period of this survey, wheat exported from elevators in the Pacific Northwest included shipments made under Public Law 480 Programs that frequently required the application of malathion to grain as it was loaded onboard ship. Also, in the latter part of 1977, several export elevators in the Gulf area were shipping wheat to countries that required treatment with malathion under conditions of the grain sale agreement. Because of equipment limitations many of the elevators involved in these markets could not apply malathion to grain after it had passed through the diverter sampler as would normally be required for grain being sampled for official grade analysis. Under an interim Federal Grain Inspection Service (FGIS) policy, these elevators were allowed to apply malathion to the grain before it passed through the diverter sampler until such time that changes in the location of malathion application equipment could be made. To account for these differences, all elevators were identified as to whether they applied malathion treatments ahead of the diverter, after the diverter or did not apply malathion to export shipments.

Bioassays

The incidence of malathion on wheat and maize obtained at export was estimated by placing 50 2-week-old adult rice weevils, *Sitophilus oryzae* (L.) (a species and stage of development particularly susceptible to even low concentrations of malathion) on 200 g portions of each sample and holding the infested samples in 0.47 l jars at $27 \pm 2^\circ\text{C}$ and $60 \pm 5\%$ r.h. Controls were placed on untreated culture wheat. After 7 days exposure, samples in which the mortality of adult weevils was greater than 20% were placed in sealed jars and held in cold storage (about 10°C) for residue analysis. Mortalities of 20% or less were considered natural and samples at this level were categorized as untreated. The rice weevil bioassays were not intended as a direct evaluation of the effectiveness of specific malathion treatments, but were used only as a rapid screening method to suggest the presence or absence of malathion residues.

Effectiveness of the malathion treatments detected by bioassay response and residue analysis was based on the incidence of live insects found in the remaining portion (about 800 g) of each sample. The insect data were developed and reported in a separate study (Storey *et al.*, 1982).

Residue analysis

Residues of malathion were extracted from the samples and cleaned for analysis in a Tracor® M.T. (Microtec) gas chromatograph by an unpublished procedure adapted by L. Davidson, Physical Science Technician, U.S. Grain Marketing Research Laboratory from that of STORRHERR *et al.* (1964) and reported in detail by QUINLAN *et al.* (1979). The lower detectable limit was 0.1 ppm.

Samples selected for residue analysis were based on the bioassay response over the range from >20 to 100% mortality, geographic location, shipping period, and the presence or absence of insects in that portion of the grain sample used for determining the incidence of insects. A total of 210 samples of wheat and 139 samples of maize were analyzed for malathion content. Selected wheat and maize samples in which no bioassay mortality was observed was also analyzed for possible malathion residues.

RESULTS AND DISCUSSION

Based on adult rice weevil bioassay mortalities greater than 20%, it was estimated that 28.4% of all wheat samples and 8.4% of all maize samples examined during the 2 year period 1977–1978 contained biologically active levels of malathion (Table 1). Malathion occurred most often in wheat from the Pacific (60.1%) and Gulf (24.8%) regions and least often in wheat from the Great Lakes (3.6%). Except for the Great Lakes Region, malathion was found more often in wheat from each region than in maize from the same region. In grain samples from the Great Lakes Region, malathion was found nearly 3 times more often in maize than in wheat.

The presence of malathion indicated by the bioassay was confirmed by gas chromatographic analysis of the residues in the grain. Average residues of malathion were 2.50 ppm (range 0.1–45) in wheat and 1.03 ppm (range 0.1–10.5) in maize and ranged from an average of 2.71 ppm in Gulf wheat to only 0.66 ppm in Pacific maize (Table 1). Except for the Atlantic Region (1.55 ppm) the average malathion residues on maize were all <1 ppm. No malathion was detected in samples that did not exhibit mortality of adult rice weevils in the bioassays.

Among all grain samples in which the presence of malathion was inferred from the bioassay data, only 2.7% of the wheat samples and 0.7% of the maize samples contained live insects (Table 1). In contrast, the overall incidence of live insects in all export samples examined during the 2 year period was 17.9% for wheat and 22.4% for maize (STOREY *et al.*, 1982). These data suggest that malathion is still sufficiently effective to limit insect infestations in grain. In most instances where live insects were present in a sample in which bioassay mortality was greater than 20% and the sample was analyzed for malathion, the residue detected was less than 1 ppm. The most frequent insect species detected in malathion-treated grain were *Rhyzopertha dominica* (E.) (lesser grain borer), *Plodia interpunctella* (Hübner) (Indian meal moth) and *Cryptolestes spp.* (flat and rusty grain beetles) and they occurred principally in samples from the Gulf Region.

Of the 79 port terminals involved in the export survey, 29 locations were identified as applying malathion before the diverter sampler and 34 locations applied malathion after the diverter. The remaining locations do not apply malathion to export shipments. Most of the Pacific and Atlantic port terminals apply malathion before the diverter. Great Lakes elevators generally apply malathion after the diverter and Gulf Locations are about equally divided before and after the diverter.

TABLE 1. INCIDENCE, CONCENTRATION, AND EFFECTIVENESS OF MALATHION IN 1977–78 WHEAT AND CORN (MAIZE) EXPORTS

Commodity and region	Number of samples	% With malathion	Mean ppm residue†	Range	% With malathion and live insects
Maize					
Atlantic	625	6.2	1.55	(0.1–10.5)	0.5
Great Lakes	580	10.5	0.94	(0.1–4.0)	0.5
Gulf	1034	8.2	0.86	(0.1–5.3)	1.1
Pacific	144	11.1	0.66	(0.1–1.3)	0
All regions	2383	(8.4)	(1.03)	(0.1–10.5)	(0.5)
Wheat					
Atlantic	83	7.2	1.23	(0.1–45)	0
Great Lakes	392	3.6	1.07	(0.1–4.4)	0.5
Gulf	777	24.8	2.71	(0.1–45)	4.5
Pacific	606	60.1	2.33	(0.1–18.8)	2.8
All regions	2058	(28.4)‡	(2.50)	(0.1–45)	(2.7)

*Based on bioassay mortality > 20%.

†Based on analysis of 139 samples of maize and 210 samples of wheat.

‡15.1% without Pacific samples.

TABLE 2. INCIDENCE AND CONCENTRATION OF MALATHION IN 1977-78 CORN (MAIZE) AND WHEAT EXPORTS FROM ELEVATORS THAT TREAT GRAIN BEFORE AND AFTER THE DIVERTER SAMPLER

Factor	No. of samples		% With malathion		Residue (ppm)			
	Wheat	Maize	Wheat	Maize	Wheat		Maize	
					Mean	Range	Mean	Range
Applications								
Before diverter	1024	707	44.4	11.6	2.81	(0.1-45.0)	1.46	(0.1-10.5)
After diverter	854	1434	11.6	7.7	1.54	(0.1-12.6)	0.75	(0.1-5.3)

In an effort to more accurately assess the incidence and concentration of malathion in wheat and maize arriving at port terminals, the malathion data from elevators that treat ahead of the diverter were compared with data obtained from those that treat after the diverter (Table 2). This comparison shows that only 11.6% of the wheat and 7.7% of the maize arriving at port terminals (treatment after diverter) contain biologically active deposits of malathion. Although decomposition of malathion residues and dilutions of treated grain with untreated grain as it moves through the marketing system are factors that would contribute to the low malathion totals, these percentages suggest that the overall use of malathion in stored-grain insect control programs from farms to export is minimal.

The relatively low concentrations of malathion in wheat (2.81 ppm) treated immediately before it passes through the diverter sampler also suggests that most port terminal elevator treatments do not result in recommended deposits of malathion on the grain. Malathion is generally applied to bulk grain at the rate of 1 pint (473 ml) of 57% premium grade malathion emulsifiable liquid in 2-5 gal. (7.6-19.0 l) of water applied to each 1000 bushels (27.2 metric tonnes) of grain. This rate of application should result in an initial deposit of between 8 and 10 ppm malathion on the grain. The problem of inconsistent and inadequate malathion deposits on port elevator treated grain has been investigated previously by the Bulk Grain Division, Agricultural Stabilization and Conservation Service in the early 1970's (unpublished data). They concluded that "one of the most common problems encountered in applying a spray to grain on a fast moving belt is that the air immediately above the grain is also moving fast and tends to wash the spray away from the grain. Consequently, much of the malathion does not contact the grain even though the spray appears to be directed on the grain". We investigated the effect of belt capacity on malathion deposition by correlating malathion residue data from elevators that apply malathion before the grain passes the diverter sampler with grain belt capacities ranging from less than 25,000 bushels (680.5 metric tonnes) per hr to 100,000 bushels (2722 metric tonnes) per hr (Table 3). Malathion residues were found to be highest at elevator locations where belt capacities were lowest, but no consistent relationship was evident between increasing belt capacities and decreased malathion residues. This suggests that factors in addition to belt capacity are involved in the low malathion deposits obtained during port terminal treatments.

TABLE 3. MEAN MALATHION RESIDUES IN 1977-1978 CORN (MAIZE) AND WHEAT EXPORTS FROM ELEVATORS WITH VARYING LOADING CAPACITIES

Loading capacity per hr (bushels)	No. of samples	Mean malathion residue (ppm)
25,000 or less	22	3.37
30-50,000	73	2.16
60-70,000	126	1.38
80-90,000	75	2.80
90-100,000	38	1.82

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